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# 16

# Fracture Toughness Testing

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## 1 INTRODUCTION: FACING A PROBLEM

A basic assumption of linear elastic fracture mechanics is the existence of a critical stress intensity factor, $K_C$, which controls the initiation of fracture. Under certain circumstances this critical value can be regarded as a materials constant, the 'fracture toughness', $K_{IC}$. The designation 'materials constant' for the fracture toughness means that, for a specific material, the danger posed by a defect in a loaded structure comprising this material is fully characterised by this single parameter. This is normally a conservative approach because $K_{IC}$ is the $K_C$-value under plane–strain conditions, that is the lowest possible $K_C$-value.

It does not seem to be necessary to point out here the high importance of the knowledge of this materials constant for technical purposes. The particular precautions which must be observed while testing in order to guarantee $K_{IC}$ to be a materials constant are well known for metals and alloys, even in cases where the material's behaviour is far from linear elastic. As a result, the procedure for measuring a $K_{IC}$-value has been standardised by the ASTM in E399-83. Ceramic materials, at least at low temperatures, can be considered to be linear elastic. This fact seems to free the experimenter from the problem, always connected with metals and alloys, of observing certain requirements in size for the specimens in order to obtain true $K_{IC}$-values from $K_{IC}$-tests. Since the grain size of a ceramic material can be neglected compared with the specimen size (which is usually true even for very small test pieces), there should be no problem in obtaining a true 'materials constant' by conducting a $K_{IC}$-test.

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Despite these convenient presumptions, $K_{IC}$-measurements on ceramic materials, even at room temperature, have produced many different results and shown up many problems. It seems necessary to standardise one or even several special procedures in order to get true $K_{IC}$-type materials constants for ceramics. To the authors' knowledge, the discussion of this problem has been initiated in the US (Quinn, G., 1987, pers. comm.) and also in Europe in Britain, France and Italy. In the FRG, the German Institute for Industrial Standardisation (DIN) held a meeting in February 1988, which was attended by one of the authors. It was announced that DIN would start its activities in this particular field in June 1988.

The aim of the present work is to point out the main problems arising in $K_{IC}$-testing with ceramic materials and to contribute the results of some specific experiments to defining a standard procedure for $K_{IC}$-testing. The present work is confined to bend tests, because in the authors' opinion these seem to be the most promising candidates for a standardised testing procedure.

## 2 STATE OF THE ART

Several problems in $K_{IC}$-bend testing with ceramic materials are already well documented. In this section these problems will be discussed first, to show the relevance of the authors' own experiments.

### 2.1 Size requirements: influence of specimen thickness

As already mentioned, ceramic materials can be regarded as ideally brittle and thus linear elastic, at least at room temperature. There should be no special specimen size requirements, especially for thickness, because unlike for metals and alloys, plane–strain conditions always exist. For example, it was shown that for an alumina containing a glassy phase at room temperature it is not necessary at room temperature to comply exactly with the ASTM E399 size requirements for bending tests, because a variation in specimen size has almost no influence on the $K_{IC}$-values (Pabst, 1972). The same author showed that a variation in the loaded volume up to a factor of 40 had no influence on the results.

A slight influence of thickness could occur when notches are used to initiate fracture instead of sharp natural cracks. This influence results from inhomogeneities in the notch root originating from the combined effect of the polycrystalline structure and the diamond grit of a saw blade. There appears to be a dependence on the 'surface structure' of the notch root similar to the dependence of the bend strength on the surface finish. An

attempt was made to modify Weibull statistics to account for the defects along the length of a notch root (Tradinik *et al.*, 1982). This statistical investigation showed that the influence of thickness on $K_{IC}$ was very small.

Summarising at this point, it appears that variations in specimen size, especially in thickness, seem to have only a small influence on $K_{IC}$-test results. Therefore, in the present investigation, the size and thickness of the specimens were kept constant.

## 2.2 Influence of different fracture-initiating defects

In $K_{IC}$-testing, critical fracture starts at an artificial 'defect'. The surface finish of the specimen, which plays a most important role in bend tests, has no influence on the results in $K_{IC}$-testing. The artificial initiating defect can be a sharp crack or a notch. The following statements concerning sharp cracks or notches for a starting defect are valid only for $K_{IC}$-testing at a high loading rate; for low loading rates there is an additional loading rate influence (see Section 2.3).

Sharp cracks can be introduced into the specimen by indentation, or in controlled growth experiments (Chantikul *et al.*, 1981; Wieninger *et al.*, 1986). When a sharp crack is used as the initiating defect for a $K_{IC}$-test, two problems arise:

—crack length measurement;
—a dependence of $K_{IC}$ on crack length ($K_R$-curve effect).

Different methods of crack length measurement have been proposed, such as direct observation during controlled loading, a side illumination technique, or the identification of the starting crack length on the fracture surface after the $K_{IC}$-test. These methods produce equivalent results (Wieninger *et al.*, 1986).

For specific materials such as coarse-grained alumina, zirconia, and for alumina with a glassy phase (the latter only at high temperatures), a dependence of the $K_{IC}$-values on the lengths of the natural cracks as starting defects has been found (Wieninger *et al.*, 1986, 1987). The symbols (●) in Figs 1 and 2 demonstrate this strong dependence.

Using notches for the starting defects, $K_{IC}$ does not depend on the depth of the notches, at least at high loading rates (symbols (○) in Figs 1 and 2). With notches, another problem arises: the dependence of $K_{IC}$ on the notch width. It was found that, in both 3- and 4-point bending, in the limit of notch width $\rightarrow 0$ (equivalent to the radius of curvature in the notch root $\rightarrow 0$), a minimum value of $K_{IC}$ may be reached (Pabst, 1972; Popp, 1981). In Fig. 3 this is shown for $K_{IC}$-measurements with alumina at room temperature (Bretfeld *et al.*, 1981). The symbols (○) are the mean values of 10 measurements, performed

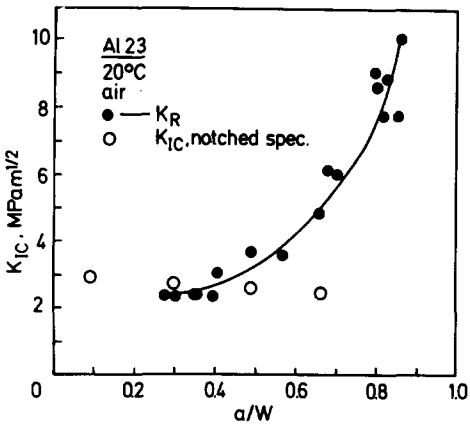



**Fig. 1.** $K_R$-curve for coarse-grained alumina at room temperature: $K_{IC}$-test at a loading rate of 1000 N/s. ○ = notched specimens (notch width 60 $\mu$m); ● = specimens with natural sharp cracks (Wieninger *et al.*, 1986).

at the Max-Planck-Institut, Stuttgart (the material is the same as that used for the present investigation). Figure 4 demonstrates this behaviour for silicon-infiltrated SiC at room temperature (Popp, 1981). Figures 1 and 2 support this point of view. The crack width necessary to get the minimum $K_{IC}$-level for the materials discussed above was $< 100\,\mu$m.

Summarising this section it may be stated that:

—with natural, sharp cracks for the starting defect for certain ceramic materials at low or high temperatures, a strong dependence on the depth of the crack may appear;

—with notches for the starting defect, the dependence on the depth of the defect vanishes, but a dependence on the width of the notch arises;

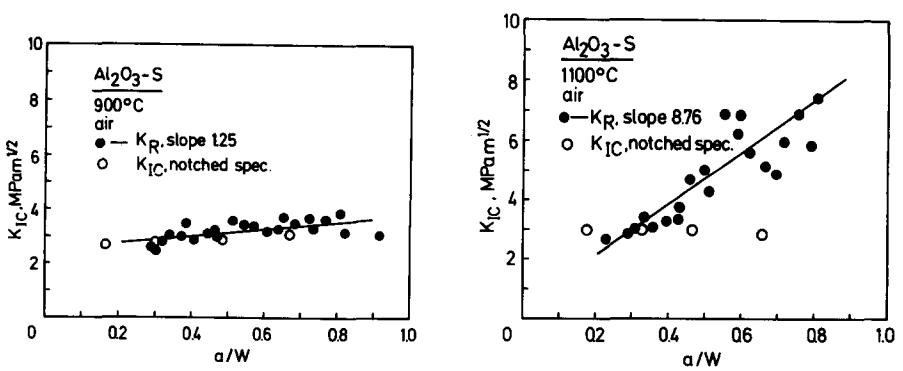



**Fig. 2.** $K_R$-curves for alumina with glassy phase: (a) at 900°C, (b) at 1100°C. $K_{IC}$-test at a loading rate of 1000 N/s. ○ = notched specimens (notch width 60 $\mu$m); ● = specimens with natural sharp cracks (Wieninger *et al.*, 1987).

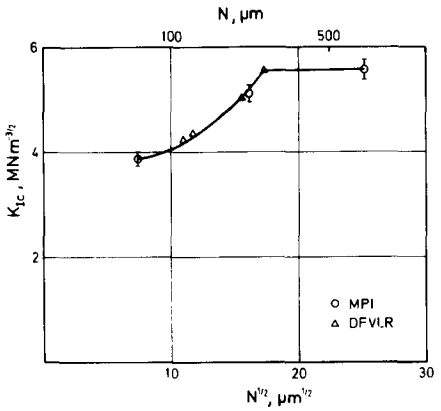



**Fig. 3.** Dependence of $K_{IC}$ on notch width: alumina at room temperature, 4-point bending, cross-head speed 0·5 mm/min. $\bar{\bigcirc}$ = notch depth to height ratio 0·2; △ = notch depth to height ratio 0·5 (Bretfeld *et al.*, 1981).

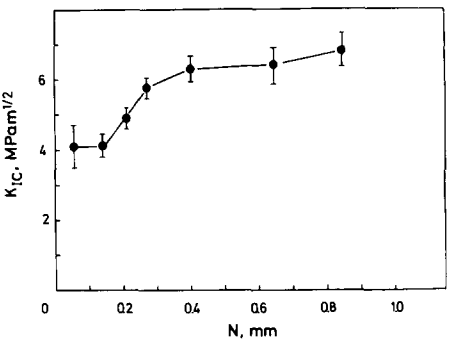



**Fig. 4.** Dependence of $K_{IC}$ on notch width: silicon-infiltrated SiC at room temperature, 4-point bending, cross-head speed 0·05 mm/min. $\bar{\bullet}$ = notch depth to height ratio 0·2 (Popp, 1981).

—with notch widths below a certain limit ($<100\,\mu$m), both the dependence on depth and on width of the starting defect seem to vanish.

In the present investigation, therefore, starting defect notches with widths of about 60 $\mu$m were used. However, it should be noted that these statements on notch width cannot necessarily be generalised for all materials, but the principles probably apply to most of them.

## 2.3 Influence of loading rate and temperature

At room temperature in air, a slight increase of $K_{IC}$-values with loading rate was found for alumina materials (Kromp & Pabst, 1981; Bretfeld *et al.*, 1981). Figure 5 shows this loading-rate dependence for pure alumina and for the same alumina with a glassy phase used in the present investigation. The reason for this dependence is subcritical crack growth in a corrosive environment, such as air, taking place prior to fast fracture at low loading rates. This subcritical crack growth is usually not accounted for when calculating $K_{IC}$.

At high temperatures for two-phase materials (alumina with a glassy phase, silicon-infiltrated SiC, and $Si_3N_4$ with sintering aids) in certain temperature ranges where the second phase changes to a viscous or plastic state, a strong dependence on loading rate appears. This dependence is demonstrated in Fig. 6 for alumina with a glassy phase at 900°C. For comparison, in the same figure the pure alumina exhibits only a slight dependence, similar to that at room temperature.

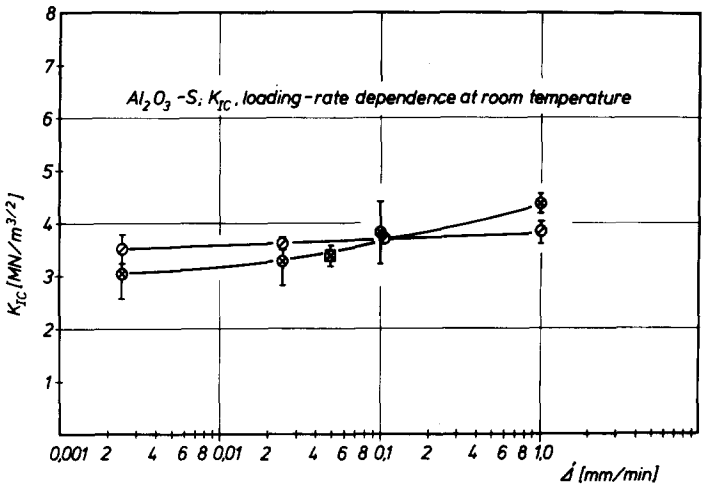



**Fig. 5.** Dependence of $K_{IC}$ on cross-head speed at room temperature. $\overline{\phi}$ = pure alumina; $\overline{\phi}$ = alumina with glassy phase, 3-point bending, notch depth to height ratio 0·2; $\overline{\phi}$ = $K_{IC}$ literature (Kromp & Pabst, 1981).

On the other hand, the state of the second phase gives rise to a strong temperature-dependence on $K_{IC}$ in the low loading rate range. Both the loading rate and the temperature-dependence are demonstrated in Fig. 7 for silicon-infiltrated SiC. Summarising this section:

—at low temperatures, subcritical crack growth and thus corrosion gives rise to a dependence on loading rate;

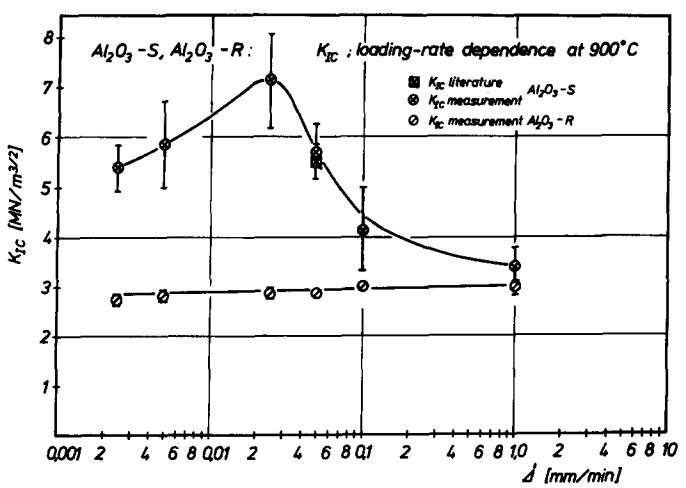



**Fig. 6.** Dependence of $K_{IC}$ on cross-head speed at 900°C. $\overline{\phi}$ = pure alumina; $\overline{\phi}$ = alumina with glassy phase, 3-point bending, notch to height ratio 0·2 (Kromp & Pabst, 1981).

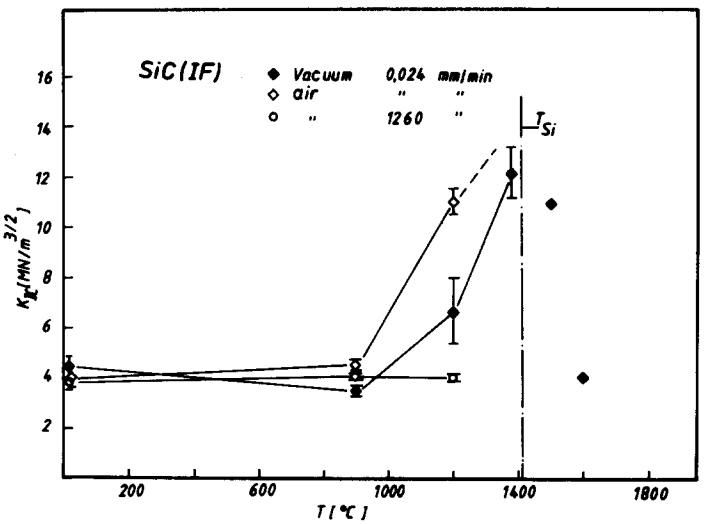



**Fig. 7.** Dependence of $K_{IC}$ on cross-head speed and on temperature for silicon-infiltrated SiC; 3-point bending, notch depth to height ratio 0·2 (Popp, 1982).

—at high temperatures, the change in the viscous or plastic state of a second phase gives rise to a strong dependence on temperature and loading rate;
—these dependences should vanish if very high loading rates are applied. This point is the subject of the present investigation.

### 2.4 Influence of loading equipment

In tests of the bend strength in 3- or 4-point bending, the size of the loaded volume has a strong influence on the result. Also, there is an influence resulting from loading on knife edges, round-ground edges ('fixed rollers'), or free-moving rollers.

For $K_{IC}$-testing, to the authors' knowledge, there is little information on this dependence. In general, any effect should not be as marked as in bend strength tests; for example, there should be no dependence on loaded volume. The only effect could be a result of friction from using fixed-edge supports instead of roller supports. This effect is also a subject of the present investigation.

## 3 EXPERIMENTS

The aim of the present investigation is to establish experimental conditions for $K_{IC}$-testing to obtain a true 'materials constant'. As already indicated,

the investigation is restricted to the bend test, which is a promising candidate for such a test procedure. Of the group of parameters that influence the test results, some are held constant and the more important ones are varied.

Taken as constant are:

—the specimen thickness $B$ and height $W$ (Section 2.1);
—the notch width and depth (Section 2.2).

The parameters which are varied are:

—the testing temperature (Section 2.3);
—the loading rate (Section 2.3);
—the testing device (Section 2.4).

### 3.1 Experimental equipment

The tests were performed in a vacuum vessel connected to a hydropulsing system, to guarantee quick response at high loading rates. The high temperature was achieved by induction heating using a $MoSi_2$ susceptor tube. The susceptor was insulated from the water-cooled induction coil by a thin-walled alumina tube. The vacuum vessel and the 4-point loading jig (see below) are shown in Fig. 8.

The experiments were performed under load-control (stress rate

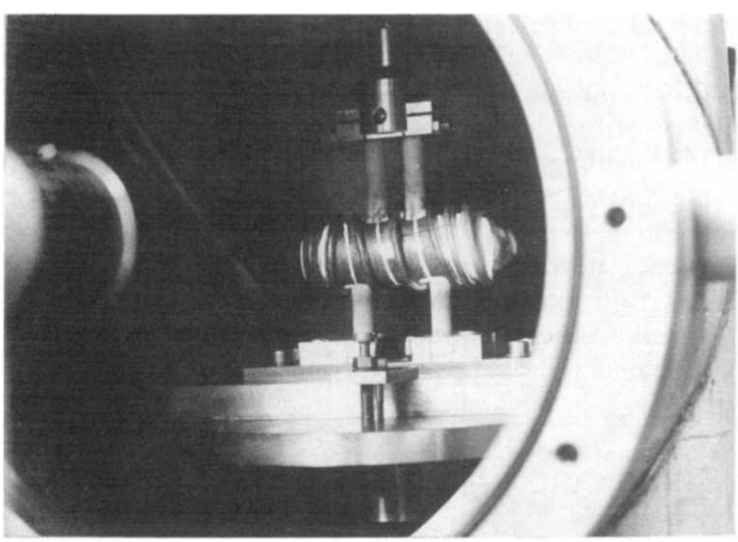


**Fig. 8.** Vacuum vessel with 4-point bending device with rounded-edge supports (radius 2·5 mm) at 1100°C in air.

$\dot{\sigma}$ = constant). The load-time relation was registered on a transient recording system. Four different loading systems were tested:

—a 3-point knife-edge support; sapphire single-crystal rods with diameter of 10 mm, machined to knife edges; span $S = 30$ mm;
—a 3-point rounded-edge support; the radius of curvature of the edges was 2·5 mm, machined from alumina rods with diameter of 10 mm; span $S = 30$ mm;
—a 4-point rounded-edge support ('fixed-roller support'); the radius of curvature of the edges was 2·5 mm, machined from alumina rods with diameter of 10 mm; outer span $S = 40$ mm, inner span $S - 2e = 20$ mm (see Fig. 8);
—a 4-point free-roller support; roller diameter = 5 mm; outer span $S =$ 40 mm, inner span $S - 2e = 20$ mm.

The complete experimental system included: vacuum vessel, hydropulsing system, induction heating system, temperature- and load-control units, transient recorder, $x$–$y$ recorder and computer.

### 3.2 Experimental conditions

An alumina with 3% wt glassy phase, a mean grain size of 10 $\mu$m, Young's modulus 360 GPa and a density of $3{\cdot}82 \times 10^3$ kg/m$^3$ were used as model material for the experiments.

The specimens had lengths of 68 or 34 mm, a thickness $B = 2{\cdot}55 \pm 0{\cdot}27$ mm and a height $W = 7{\cdot}0 \pm 0{\cdot}25$ mm. They were notched to a notch length to height ratio $a{:}W = 0{\cdot}17 \pm 0{\cdot}03$ by means of a diamond-covered copper blade with a thickness of 50 $\mu$m, resulting in a notch width of about 60 $\mu$m.

Experiments were performed at room temperature and at 1100°C. The inertia of the inductive heating system being very low, the level of 1100°C was reached in 5 min.

A cross-head speed of the order 1 mm/min is usually chosen for technical convenience in $K_{IC}$-testing. With displacement and span for the individual specimen size and material used in this investigation, a corresponding stress rate of $\dot{\sigma} = 35$ MPa/s† was calculated. This relatively low stress rate was applied in the present experiments in order to allow a comparison of the results with those from other investigations.

† The calculated stressing rate is based on the rate of application of load to a solid beam showing the same flexural characteristics as the notched beam in question, and is thus a purely nominal rate unrelated to the acutal stress field in the specimen.

It should be emphasised here that an experiment performed at a constant cross-head speed is an uncontrolled experiment, for neither load nor displacement are controlled.

Additionally, a very high stress rate of $\dot{\sigma} = 10\,000$ MPa/s was chosen to rule out all effects that arise at the low stress rates (see Section 4). The stress rates were individually calculated for each specimen because the dimensions of the specimens varied by a certain amount as stated above.

Seven experiments were performed for each test condition; the mean and maximum deviations of these groups of seven experiments are presented. For the calculation of the $K_{IC}$-values, three different geometric correction functions $Y(a:W)$ were employed: Srawley (1976), Steigerwald (1970) and Brown & Srawley (1966). The results were almost identical; the first (Srawley, 1976) was generally used for the further calculations.

## 4 EXPERIMENTAL RESULTS AND DISCUSSION

### 4.1 Influence of the testing device on $K_{IC}$-results

At first, experiments with the four different loading jigs were performed at 20 and 1100°C. To attempt to rule out the expected influence of corrosion at 20°C and the influence of the second phase at 1100°C, the high stress rate of $10^4$ MPa/s was applied in both sets of experiments. Figure 9 shows the results. It is obvious that the levels of $K_{IC}$-values are different for 20°C ($K_{IC} = 3{\cdot}81$) and 1100°C ($K_{IC} = 2{\cdot}97$). This decrease with increasing temperature was expected.

The 3-point knife edge, and the 3- and 4-point rounded-edge support systems gave nearly identical results at room temperature. Only the 4-point free-roller system led to a 5% lower value (Fig. 9). This result could also be expected, because fixed-support systems give rise to friction between test specimen and support, and thus to higher sustained loads prior to fast fracture.

Unfortunately, a free-roller support for high temperatures was not available. Therefore, this friction effect could not be demonstrated at 1100°C. Only the fixed systems could be tested at the high temperature, but the 4-point rounded-edge support system obviously produced higher friction than the 3-point systems (Fig. 9).

Additionally, at the high temperature the influence of a misalignment was tested for the 4-point rounded-edge support. One of the inner support edges

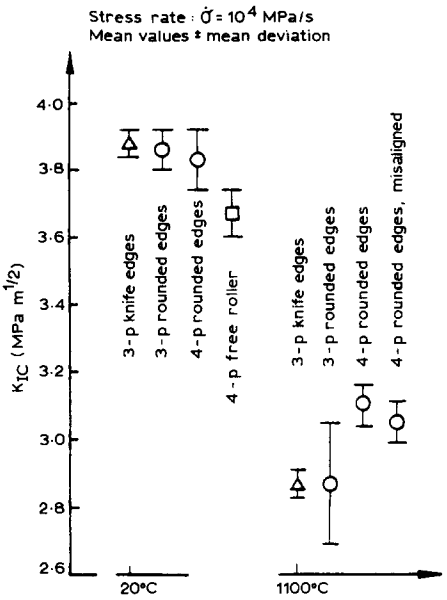



**Fig. 9.** Influence of the specific testing device on $K_{IC}$-results. Stress rate: 10 000 MPa/s, mean values ± mean deviation.

was turned by 20° in the plane of the specimen. The influence of such a misalignment was very small (Fig. 9).

Summarising this section:

—3- or 4-point testing devices give equivalent results, which would not be true for bend strength measurements;
—friction effects have an influence on the results: systems with fixed supports show higher values than systems with free rollers. At high temperatures for the systems with fixed supports, the 4-point systems show higher values than the 3-point systems.

It should be mentioned here that the free-roller system was not free to pivot about an axis parallel to the length of the specimen—this point needs to be investigated separately. If the specimens are well machined, i.e. the surfaces are plane and parallel, then in the authors' opinion the absence of ability to pivot should have little influence on the result.

All these deviations are less than 10%. If one recognises the scatter of the measurements for the particular testing jigs (the maximum deviations for the particular devices are shown in Fig. 10), it could be stated that for $K_{IC}$-testing, the influence of a specific loading device on the results is not significant.

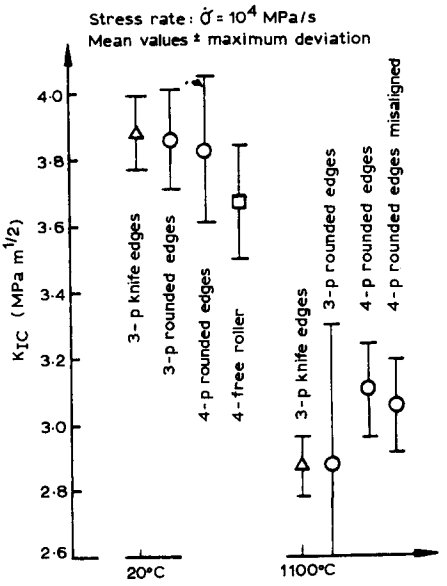



**Fig. 10.** Influence of the specific testing device on $K_{IC}$-results. Stress rate: 10000 MPa/s, mean values ± maximum deviation.

Concluding this section, it is found that the 3-point knife-edge support system shows the smallest scatter, especially at the high temperature. It may well be that this system is the best for defining the test conditions (see Fig. 10).

## 4.2 Influence of loading rate and temperature on $K_{IC}$-results

As already discussed in Section 2.3, the loading rate and the testing temperature mutually influence the results of $K_{IC}$-testing. The following measurements were all performed with the 3-point rounded-edge supports. To clarify the influence of water-vapour corrosion, measurements in air were compared with measurements in a vacuum of $5 \times 10^{-3}$ Pa.

At the high stress rate of $10^4$ MPa/s, the results of the measurements in air and in vacuum at 20°C were identical. At 1100°C, the result for the vacuum measurement was at a slightly higher level compared to the test in air, but the scatter bands of both tests overlapped (Fig. 11). Both vacuum results at the very high stress rate were about the same as the measurements in air already shown in Fig. 9.

The low stress rate of $\dot{\sigma} = 35$ MPa/s, corresponding to the loading rate commonly used, gave a variety of results. For this low rate, the result in the corrosive environment at room temperature was distinctly lower (15% less),

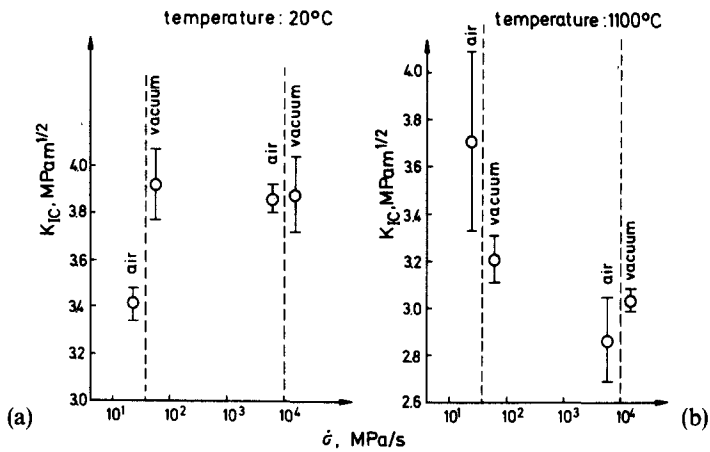


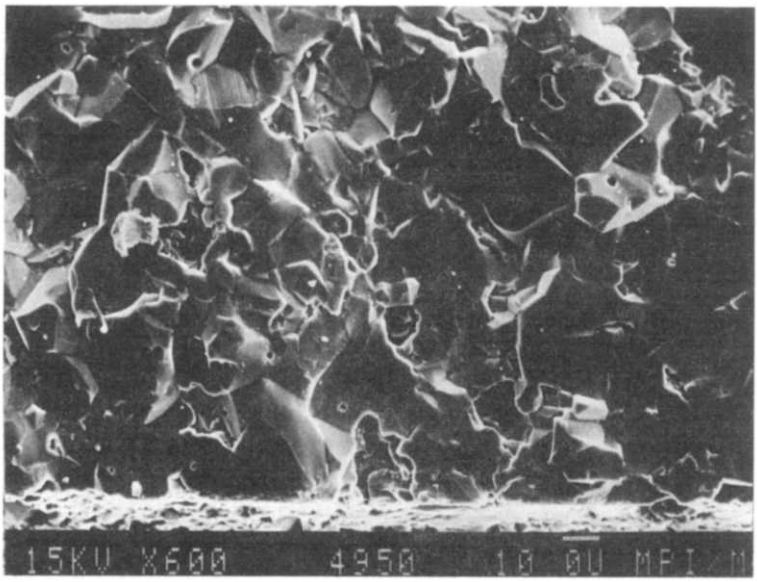

**Fig. 11.** Influence of loading rate on $K_{IC}$-results (stress rates of 35 and 10 000 MPa/s) in air and in vacuum (mean values ± mean deviation): (a) at room temperature, (b) at 1100°C.

while the value in vacuum was at about the same level as the high stress rate result (Fig. 11). Only subcritical crack growth prior to fast fracture could be the cause for this deviation in air. From the fracture surfaces, this fact could hardly be detected. In general, the fracture surfaces for the low stress rate in an air environment appear more intergranular than those of the specimens broken at the high stress rate (compare Figs 12(a) and (b)).

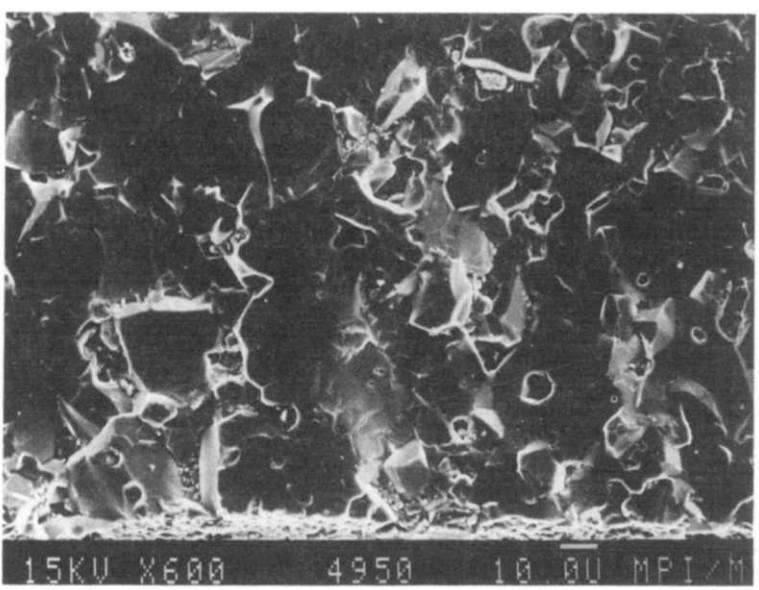
At 1100°C, the results for the low stress rate were higher than those for the high stress rate (Fig. 11): this fact was noted previously (see Fig. 6). Subcritical crack growth, which at these temperatures can only result from thermally activated processes, does not play an important role. This high level of $K_{IC}$-values at the low stress rate may result from the blunting of microcracks by the viscous flow of the glassy phase. Viscous flow is energy-absorbing and results in a higher $K_{IC}$-level. This can be observed directly on the fracture surfaces; an example is given in Fig. 13. The glassy phase covers the grains and moves to triple junctions, thus blunting microcracks, which start predominantly from these positions (see especially Fig. 13(b), at the lower left of the picture).

To summarise this section:

—at low loading rates and temperatures, corrosive subcritical crack growth takes place and reduces the level of the $K_{IC}$-values;
—at low loading rates and high temperatures, energy-absorbing effects caused by the low-viscosity state of the second phase occur and thus raise the $K_{IC}$-values;
—at very high loading rates, these effects do not occur.

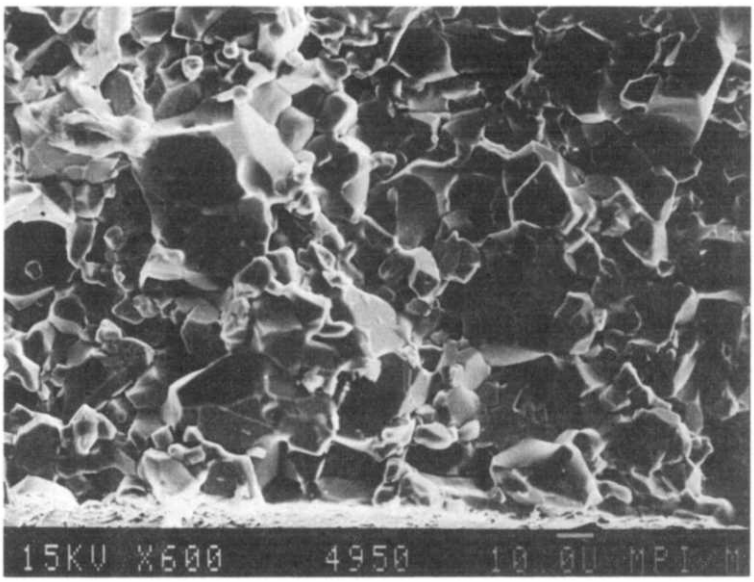



(a)

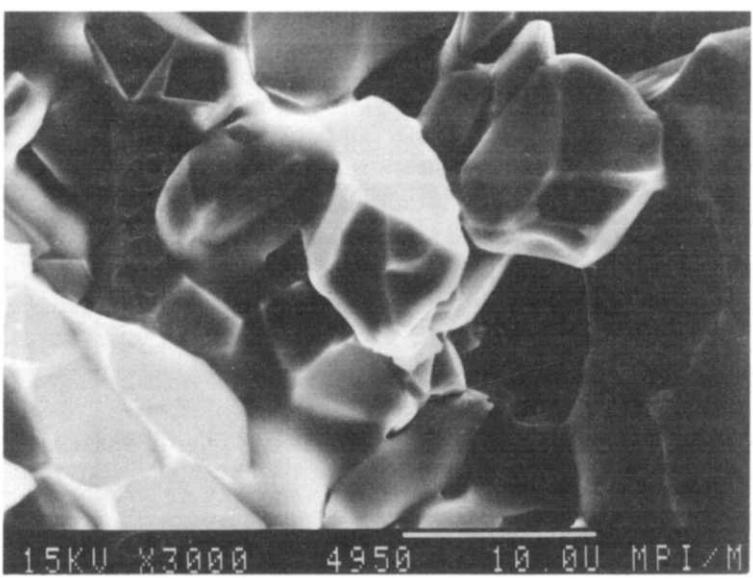



(b)

**Fig. 12.** Fracture surfaces at different stress rates (a) 35 MPa/s, and (b) 10 000 MPa/s; room temperature in air. The notch root appears at the foot of the images.




(a)




(b)

**Fig. 13.** Fracture surface: stress rate 35 MPa/s at 1100°C in vacuum. The notch root appears at the foot of the images. Image (b) shows detail from image (a).

## 5 SUMMARY AND CONCLUDING REMARKS

The bending method is a promising candidate for a standard $K_{IC}$-testing procedure. First, the influences of the specimen dimensions, the fracture-initiating defect, the loading rate, the temperature and the use of specific loading fixtures were discussed. Then, experiments were presented using an alumina with 3% wt glassy phase as a model material to clarify the influence of a specific loading jig: i.e. 3-point bending with knife-edge supports; 3- and 4-point bending with rounded-edge supports ('fixed-roller supports'); and 4-point bending with free-roller supports. Additionally, the influence of two extremely different loading rates (stress rates of 35 MPa/s and 10 000 MPa/s) and two different temperatures (20 and 1100°C) was elucidated by the experimental results.

The following conclusions for the establishment of a testing procedure for the measurement of $K_{IC}$ as a 'materials constant' can be drawn, provided that a notch of width $< 100\,\mu m$ is used for the starting defect:

—with different loading fixtures, the results differ by only a few per cent; therefore a 3-point bending system with knife-edge supports would be suitable, because it is easier to use compared with 4-point free-roller systems, especially at high temperatures;
—the commonly applied loading rates (cross-head speeds) in the range of 1 mm/min are to be avoided, because they produce conditions where the influences of corrosion at low temperatures and of a second phase at high temperatures is greatest:
—very high loading rates should be applied, because with these the influences of corrosion and second phases vanish.

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